

# PATENT SPECIFICATION

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## (54) HYDRAULIC FLUIDS AND THEIR PREPARATION

(71) We, SHELL INTERNATIONAL RESEARCH MAATSCHAPPIJ N.V., a company organized under the laws of the Netherlands, of 30 Carel van Bylandtlaan, The Hague, the Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to hydraulic fluids, particularly brake fluids, comprising (a) a lubricating component containing a polyoxyalkylene fluid and (b) a diluent component; the invention also relates to the preparation of such fluids.

Hydraulic fluids containing more than 20%, by weight, of a polyoxyalkylene fluid as lubricating component and at least 10%, by weight, of an ether of triethylene glycol are already generally known. Such fluids, however, no longer fulfil the stringent specifications imposed on modern brake fluids suitable for disc brakes.

In order to satisfy these more stringent requirements, it has often been necessary to form brake fluids from a large number of components. Surprisingly, it has now been found that such hydraulic fluids which completely satisfy modern requirements can be formed from only two basic components, whereby good lubricating properties together with a relatively low viscosity and low volatility are obtained, without there being a substantial adverse effect on the rubber swell properties.

According to the present invention a hydraulic fluid comprises (a) a lubricating component containing a polyoxyalkylene liquid and (b) a diluent component containing a mixture of mono-isopropyl ethers of tri-, tetra- and penta-ethylene glycols.

Although component (b) can be prepared by mixing the respective mono isopropyl ethers, component (b) is preferably prepared as a distillation fraction of the reaction pro-

duct of isopropyl alcohol and ethylene oxide, as described below.

Component (a) preferably has a molecular weight between 180 and 6000, in particular between 200 and 3000. Suitable components are condensation products of ethylene oxide and/or propylene oxide, including block copolymers and the corresponding mono- and diethers or esters with, for example, aliphatic alcohols or aliphatic carboxylic acids. Especially at low molecular weights the non-etherified and non-esterified products are preferred for their better lubricating properties.

The proportion of component (a) is preferably between 5% and 30%, by weight, and particularly about 20%, by weight, based on the mixture of (a) and (b).

The composition of component (b) may vary depending on the desired high-temperature properties. Thus component (b) may largely consist of a mixture of mono-isopropyl ethers of tri- and tetra-ethylene glycol and only relatively small amounts of the mono-isopropyl ether of penta-ethylene glycol. For extreme high-temperature requirements component (b) may largely consist of a mixture of mono-isopropyl ethers of tetra- and penta-ethylene glycol and only relatively small amounts of the mono-isopropyl ether of tri-ethylene glycol. Small amounts of mono-isopropyl ethers of lower and/or higher polyethylene glycols, in particular hexa-ethylene glycol and the mono-isopropyl ether of mono-ethylene glycol and/or non-etherified molecules and/or di-isopropyl ethers may be present. The amounts of each of these compounds are generally less than 30%, by weight, of component (b), while the total amount of these compounds is generally less than 50%, by weight, of component (b). The amount of component (b) is preferably between 95% and 70%, by weight, in particular 80%, by weight, based on the mixture of (a) and (b).

Since component (b) functions as a diluent

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for component (a), the viscosity of component (b) should of course be lower than that of component (a). It is clear that this viscosity is not only dependent upon the molecular weight, but also upon the molecular structure. The molecular weights of components (a) and (b) may therefore partially overlap one another.

Small amounts of other components such as mono- and di-C<sub>1-4</sub> alkyl ethers of mono- and polyethylene glycol being different from components (a) and (b) or of mono- and polypropylene glycol, or of mono- or polyglycols may be present. The proportions of these may be, for example, up to 30%, by weight, of (a)+(b).

Small amounts, for example, between 0.1% and 10%, by weight, of additives, such as anti-corrosion, anti-oxidation and anti-wear additives may be incorporated.

Suitable anti-oxidation additives are alkylphenols, alkylated bisphenols and compounds such as diphenylol-propane, in particular 2,2-di-(4-hydroxyphenyl)propane.

Suitable anti-corrosion and anti-wear additives are alkali metal salts of fatty acids with 9—22, in particular 14—18 carbon atoms, such as sodium and potassium laurate, stearate and oleate.

Component (b) is preferably prepared by passing an excess of isopropyl alcohol and ethylene oxide, preferably in the liquid phase, through a reactor in the presence of a catalyst and at elevated pressure and temperature.

The reaction product is fractionated, the fraction containing component (b) is separated, and excess isopropyl alcohol and any mono-isopropyl ethers of mono- and/or diethylene glycol and, if desired, triethylene glycol formed, is at least partially recirculated. Preferably after fractionating off the isopropyl alcohol, a bottom product including the catalyst, any salts formed and undesirable residual components is separated in a flash distillation column. Component (b) thus obtained is substantially or completely colourless, and from the bottom fraction with a higher boiling range than component fraction (b) material can, if desired, be removed for use as component (a) or as a constituent thereof.

The catalyst is preferably an alkaline catalyst, such as sodium hydroxide or potassium hydroxide. The reactor is preferably tubular. The reaction is preferably carried out between 100° and 200°C and between 5 and 100 atmospheres, in particular between 120° and 180°C and 15 and 25 atmospheres. The starting temperature of the reaction mixture is preferably between 100° and 170°C and the final temperature is preferably between 130° and 200°C. The reaction is preferably carried out continuously.

The present invention is illustrated by the following example:

#### EXAMPLE

A component (b) was prepared in the following manner. The reactor was a tube with good heat insulation and had a diameter of 30 cm and a length of 225 m. The pressure in the reactor was 16 atg. To the tube were added every hour 8.1 kmol (485 kg) of isopropyl alcohol, 158.6 kmol (9515 kg) of recirculated isopropyl alcohol, 9.8 kmol (430 kg) of ethylene oxide, 0.67 kmol (100 kg) of recirculated mono-isopropyl ether of diethylene glycol and 0.041 kmol (1.6 kg) of NaOH as a 4.5% by weight solution in 0.23 kmol (34.5 kg) of recirculated mono-isopropyl ether of diethylene glycol. The temperature at the beginning of the tube was raised to 145°C by pre-heating it with steam and the temperature at the end of the tube was 165°C. The reaction product was separated by fractional distillation partially at reduced pressure into 158.6 kmol/hour (9515 kg/hour) of unconverted isopropyl alcohol, 5.3 kmol/hour (550 kg/hour) of mono-isopropyl ether of monoethylene glycol, 1.3 kmol/hour (187 kg/hour) mono-isopropyl ether of diethylene glycol, of which 0.90 kmol/hour (134.5 kg/hour) was recirculated, 252 kg/hour of a fraction with a boiling range between 255° and 350°C (converted to normal pressure) being the desired component (b) and 70 kg/hour of bottom product containing, inter alia, the catalyst and mono-isopropyl ethers of higher polyethylene glycols. The composition of 252 kg of component (b) was as follows:

	11 kg	of mono-isopropyl ether	of diethylene glycol	
	97 kg	" "	" "	triethylene glycol
	68 kg	" "	" "	tetraethylene glycol
	34 kg	" "	" "	pentaethylene glycol
105	42 kg	" "	" "	higher polyethylene glycols

The properties of the component (b) thus prepared and of mixtures of this component (b) with a component (a) according to the invention are shown in the following table

and are compared with the requirements according to SAE specification J 1703 A for brake fluids.

TABLE

Mixture Properties	Component (b)	78% (b) 22% (a)	90% (b) 10% (a)	87% (b) 13% (a)	80% (b) 20% (a')	SAE J1703A
Boiling point, °F	509	507	—	—	—	>374°F
Viscosity -40°C, cS	600	3000	1410	1730	1600	<1800
Rubber swell 70 hours, 120°C, mm	1.16	0.99	—	—	0.6	0.15-1.4
Hardness	-11	-11	—	—	-8	0- -15
Residue	—	—	—	pass	—	traces only
Evaporation % by weight	—	—	—	58.4	—	<80
Pour point residue °C	—	—	—	<-5	—	<-5

## In this Table

Component (a) is:

- 5 adduct of diethylene glycol and mixture of ethylene oxide and propylene oxide (50/50, by weight), molecular weight 2600;

Component (a') is:

- 10 polyethylene glycol, molecular weight 200; whereas all mixtures of (a) or (a') and (b) contain 1.5%, by weight, of potassium oleate and 0.4% by weight of diphenylol propane.

- 15 This Table shows the good properties of the hydraulic fluids according to the invention. Moreover, these fluids exhibit no, or substantially no, significant corrosion of various metals, such as steel, aluminium and copper, and they exhibit good water tolerance. In spite of the presence of isopropyl groups, the rubber swelling characteristics satisfy stringent requirements. The good low-temperature properties are surprising. For example, it has been found that the mono-isopropyl ether of tetraethylene glycol has a viscosity of 1000 cS at -40°C, whereas the corresponding monoethers of methyl, ethyl and n-butyl alcohol are solid at -40°C.

- 20 The present invention also includes a hydraulic system containing a hydraulic fluid according to the invention. The present invention also includes a process for the preparation of a hydraulic fluid as defined above, which comprises forming a blend of (a) a lubricant component containing a polyoxy-alkylene liquid and (b) a diluent component

prepared by passing an excess of isopropyl alcohol and ethylene oxide through a reactor in the presence of a catalyst at elevated temperature and pressure, and separating from the reaction product by fractionation a mixture of mono-isopropyl ethers of tri-, tetra- and penta-ethylene glycols, unreacted isopropyl alcohol and any mono-isopropyl ethers of mono and/or diethylene glycol formed being at least in part recirculated to said reactor.

## WHAT WE CLAIM IS:—

1. A hydraulic fluid comprising (a) a lubricating component containing a polyoxy-alkylene liquid and (b) a diluent component containing a mixture of mono-isopropyl ethers of tri-, tetra- and penta-ethylene glycols.

2. A fluid as claimed in claim 1, wherein the component (a) has a molecular weight between 180 and 6000.

3. A fluid as claimed in claim 1 or claim 2, wherein the component (a) is a condensation product of ethylene oxide and/or propylene oxide.

4. A fluid as claimed in any one of claims 1-3, wherein the proportion of component (a) is 5% to 30%, by weight.

5. A fluid as claimed in any one of claims 1-4, wherein component (b) consists largely of a mixture of the mono-isopropyl ethers of tri- and tetra-ethylene glycols.

6. A fluid as claimed in any one of claims 1-5, wherein additives are present.

7. A hydraulic fluid as claimed in claim 1 and substantially as hereinbefore described.

8. A process for the preparation of a

- hydraulic fluid as claimed in claim 1, which comprises forming a blend of (a) a lubricant-component containing a polyoxyalkylene liquid and (b) a diluent component prepared by
- 5 passing an excess of isopropyl alcohol and ethylene oxide through a reactor in the presence of a catalyst at elevated temperature and pressure, and separating from the reaction product by fractionation a mixture of mono-
- 10 isopropyl ethers of tri-, tetra- and penta-ethylene glycols, unreacted isopropyl alcohol and any mono-isopropyl ethers of mono- and/or diethylene glycol formed being at least in part recirculated to said reactor.
- 15 9. A process as claimed in claim 8, wherein an alkaline catalyst is used.
10. A process as claimed in claim 9, wherein the alkaline catalyst is sodium hydroxide.
- 20 11. A process as claimed in any one of claims 8—10, wherein the reaction mixture is passed continuously through a tubular reactor.
- 25 12. A process as claimed in any one of claims 8—11, wherein the reaction is carried out at a temperature between 100° and 200°C and at a pressure between 5 and 100 atmospheres.
13. A process as claimed in claim 12, wherein the reaction is carried out at a temperature between 120° and 180°C and at a pressure between 15 and 25 atmospheres.
- 30 14. A process as claimed in any one of claims 8—13, wherein after fractionating the excess isopropyl alcohol a catalyst-containing bottom product is separated in a flash distillation column.
- 35 15. A process as claimed in claim 14, wherein the bottom fraction remaining after fractionation of fraction (b) is used as component (a) as claimed in claim 1, or as a component thereof.
- 40 16. A process as claimed in claim 8 and substantially as hereinbefore described with reference to the Example.
- 45 17. A fluid prepared by the process claimed in any one of claims 8—16.
18. A hydraulic system containing a fluid as claimed in any one of claims 1—7 or 17.
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